

# National Bureau of Standards



November 1956  
Volume 40  
Number 11

## Technical News Bulletin

NOV 1956

### MOTION PICTURES OF METAL FATIGUE

#### automatically record details

**T**HE NATIONAL BUREAU OF STANDARDS has recently constructed a small fatigue-testing machine equipped with a motion picture camera for filming the microscopic features of a metal surface during fatigue fracture. Designed by J. G. Weinberg of the Bureau's mechanical metallurgy laboratory, the apparatus uses a clock-controlled, 16-mm camera to take time-lapse motion pictures of aluminum specimens under torsional stress.

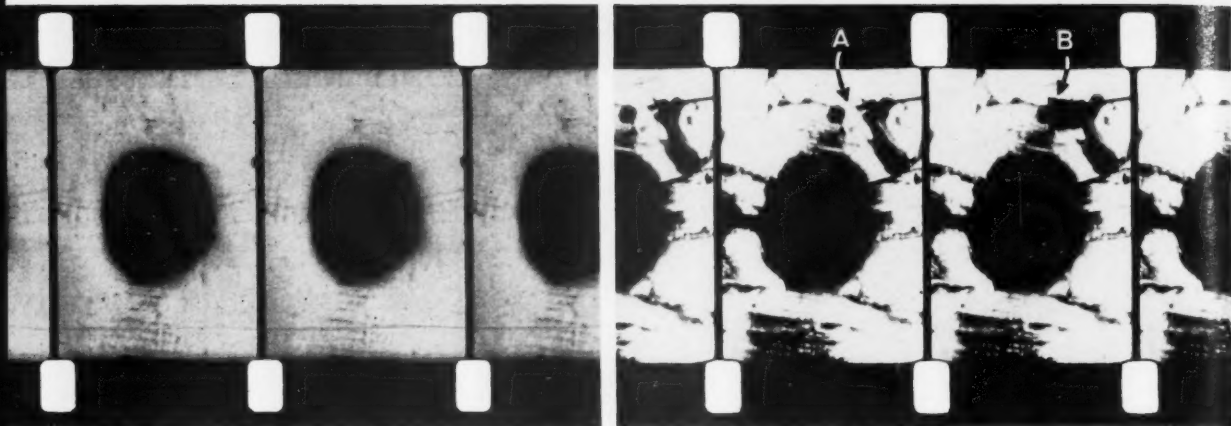
An understanding of fatigue phenomena is important to the engineer in predicting the life of a metal in service and to the metallurgist in designing more fatigue-resistant metals. In order to obtain detailed information on fatigue for use in aircraft design, the National Advisory Committee for Aeronautics is sponsoring a Bureau study of the basic factors influencing fatigue crack initiation. The films made in the course of this investigation should simplify tedious laboratory determinations by providing automatically recorded, detailed pictures of the fatigue process.

The base of the fatigue-testing machine is a block of steel designed to fit the stage of a metallurgical microscope. The base is machined at the top to a shallow U-shape, and a horizontal collet for holding the test specimen is inserted through the upper parts of the block. Two small eccentric cams are mounted at the ends of a motor-driven shaft passing through the lower part of the base so that cam followers can transmit the

load to the ends of the collet above. The metal specimen to be filmed is pressed into the collet, and an alternating torsional load is applied through the cam followers by the cams, which are mounted 180° out of phase. The amount of load is determined by the degree of cam eccentricity. A 0.01-in. hole, drilled in the top surface of the specimen, provides a focusing target for the microscope. Any fatigue failure that occurs will tend to develop around the hole and thus be in the field of the microscope.

The camera is attached to the principal eyepiece of the microscope, and a second eyepiece permits simultaneous visual observation. A large disk with a hole near its edge revolves on the motor shaft and serves as a shutter by interrupting the light to the microscope's vertical illuminator. This arrangement permits successive exposures at the same point of the stress cycle. It is necessary because each frame may be exposed a number of times before another frame takes its place.

The film is advanced at regular intervals by a synchronous motor-driven cam that actuates the camera's single frame button, the camera shutter being adjusted to close only during film transport. Because the speed of the fatigue machine can be adjusted with respect to the movement of the film, the number of stress cycles per frame can be varied depending on how rapidly changes are taking place in the surface of the metal.

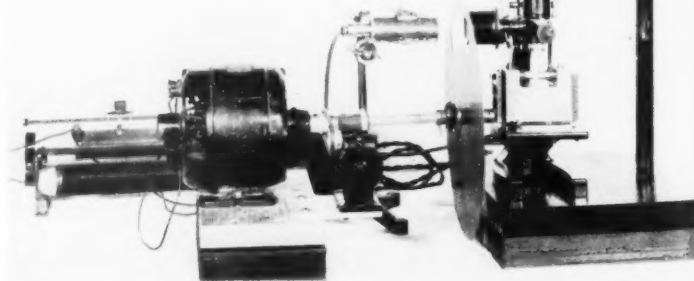
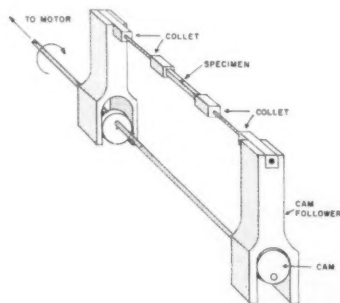


Frames from motion picture showing the early and later stages of aluminum under torsional stress. In the early stages (left) slip bands developed in the highly stressed regions around a 0.01-in. hole that had been drilled in the metal surface. *Right:* Later some of the slip bands developed into cracks, and a material of unknown composition was extruded from the crack at irregular intervals. Note difference in amount of dark material at A and B. Each frame was exposed for approximately 25 cycles of stress.

Portions of the films taken during the early stages of the fatigue test showed no unexpected changes. Slip bands developed in the highly stressed regions around the hole, and these gradually became more numerous and heavier until some of them developed into cracks. During the latter stages of the test a crack developed in an area that had contained no slip bands in the early part of the test. A short time later material began to be extruded from this opening at irregular intervals, but in surprisingly large quantities considering the

size of the crack. As the crack was only 0.01 in. long and the extruded ribbon very thin, the actual amount of material driven out was extremely small. This phenomenon occurred suddenly and was not observed until the film was developed and viewed, so that no attempt could be made to collect the extrusion for chemical analysis. However, it has been suggested that the extruded material, if not an oxide of some kind, is so fine that it probably reacts immediately with the atmosphere to form one.

The fatigue-testing machine is set on the stage of a metallurgical microscope (right). The clock-controlled, 16-mm camera, set on top of the microscope, takes time-lapse motion pictures of aluminum specimens under torsional stress. An alternating torsional load is applied by cams mounted 180° out of phase on the motor-driven shaft. Schematic shows the moving parts of fatigue-testing machine, indicating the arrangement of motor shaft, cams, cam followers, collet, and test specimen.



# APPARATUS FOR ETCHING FINE POINTS

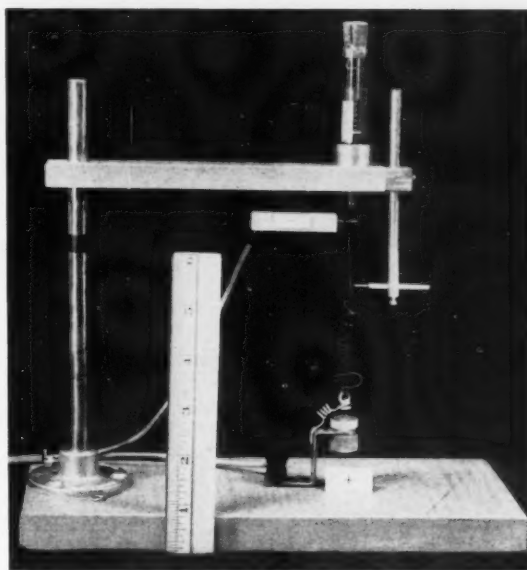
**A**N IMPROVED METHOD for producing fine points by electrolytic etching of wire under tension has been developed. Although these points were constructed for use as electron sources for a field emission X-ray microscope, they could also be used as field-emission ion sources to replace the thermionic emitters in accelerators and mass spectrographs. The improved process was developed by R. C. Placious and R. A. Schrack of the Bureau's electron physics laboratory as part of a project for the Air Research and Development Command.

The apparatus is similar to that developed by Niemeck and Ruppig for the electrolytic etching of points under tension, but improves the original design by employing a tensiometer to preset tension to a known value. With the accurate determination of tension, control of point size and geometry becomes possible. A high tension causes the wire to break in a shorter time and produces a larger point than a low tension with a longer etching period. Current-voltage characteristics of the points as field emitters can be quite accurately predicted on the basis of the tension applied during etching, because the size of the point determines the amount of current emitted at a particular voltage.

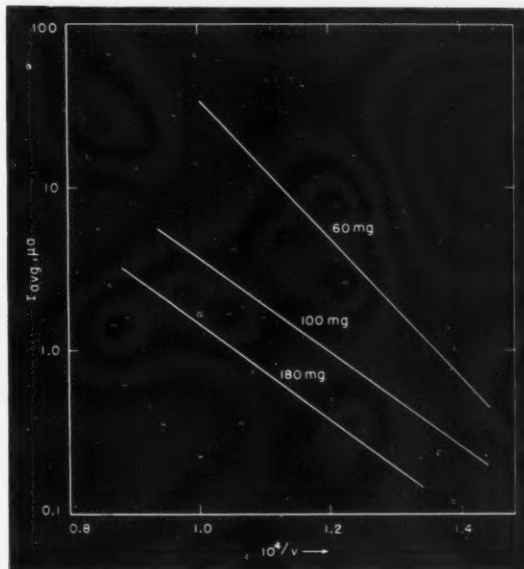
A tensiometer consisting of a fine wire spring in a calibrated glass tube is mounted on a vertically adjustable arm. To preset the tension, one end of the calibrating spring is temporarily fastened to a tension spring mounted on the apparatus directly below the tensiometer. The desired tension on the tensiometer spring is obtained by adjusting both the vertical position of the arm and the extension of the tension spring. A pointer clamped to the arm marks the point of contact between the calibrating and the tension springs at the final tension adjustment. Also attached to the arm is a nickel wire loop containing a film of electrolytic solution. The point is formed where the wire passes through the film.

When the desired tension has been obtained, the tensiometer is replaced by a chuck holding a metal shank to which the wire to be etched is welded. A crosspiece spotwelded to the metal shank positions the wire in the etching plane and also serves as a stop for locating the shank in the chuck. After the point has been formed this crosspiece will locate it in the field emission equipment with minimum adjustment. Attaching the tension spring to the wire at the position indicated by the pointer places the wire under the same tension set by the tensiometer.

It is important to clean the wire before etching, and isolating the equipment from vibration and air currents aids in obtaining symmetrically etched points. Any size wire can be used but the Bureau's points were etched from 5 or 6 mil diameter tungsten wire in a 0.6 normal sodium hydroxide electrolyte. The electrolyte is inserted with an eyedropper into the nickel wire loop through which the tungsten wire passes, and 8 to 10 v dc is applied so that the conventional direction of current flow is from the wire to the loop. Vary-



Apparatus for producing fine points by the electrolytic etching of a wire under controlled tension. After the tensiometer presets the tension, it is replaced by a shank holding the wire to be etched. The wire passes through a nickel loop in which an electrolyte is inserted to form the point when current is allowed to flow. Below: Graphs of current versus  $10^4/V$  voltage obtained in field emission tests of tungsten points electrolytically etched under 3 different tensions (in milligrams). Smaller tensions yield larger currents for a given voltage because they permit the electrolytic process to continue longer and so form finer points, which produce more concentrated fields. Slope of graph depends on such characteristics as smoothness and shape of points.



ing the amount of electrolyte in the loop controls this current; it is initially adjusted to 2 ma for tungsten and drops to about 0.5 ma just before breaking. Etching takes about 15 min. The current values and time are not critical.

For further technical details, see Apparatus for etching fine points under controlled conditions, *R. A. Schrack and R. C. Placius, Rev. Sci. Instr.* **26**, 412 (June 1956). Also see Elektrolytische Atzung von Kathodenspitzen für das Feldelektronenmikroskop, *F. W. Niemeck and D. Rupp, Z. angew. Phys.* **6**, 1 (1954).

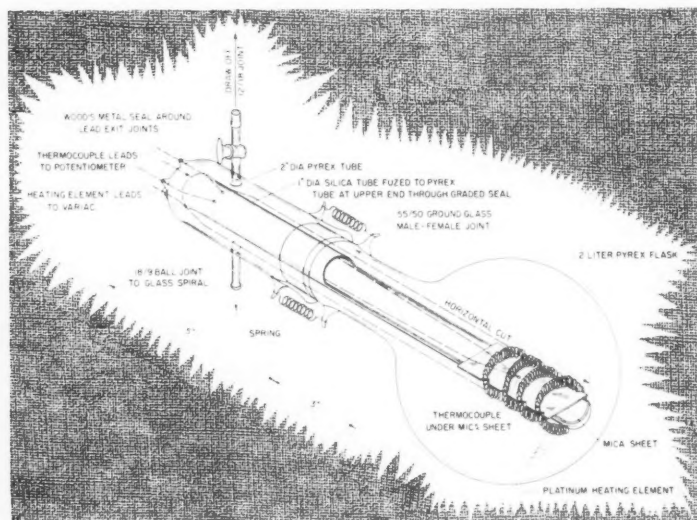
## Controlled Burning of Combustible Materials

A SIMPLE, RAPID METHOD has been developed for burning combustible materials under closely controlled conditions. With this procedure and equipment, the gaseous products formed when organic materials are burned in air can readily be collected and analyzed.<sup>1</sup> The results give a quantitative estimate of the combustion gases produced from such organic coatings as paints, asphalts, and plastic compounds. This information is useful in selecting organic coatings with particular thermal breakdown properties for use in buildings and other structures. The method was developed by A. Schriesheim, of the Bureau's floor, roof, and wall coverings laboratories, working under the sponsorship of the U. S. Air Force.

of the ratio between air volume and specimen weight at any initial firing temperature up to 550° C.

The Bureau's equipment for burning organic materials consists essentially of a combustion chamber containing a heating element for firing the specimen, and apparatus to control the amount of air in the chamber. The combustion chamber, a two-liter Pyrex flask, can be tilted so that the specimen will slide down a silica tube inside the flask into the heating element.

In use, a porcelain boat containing the specimen is placed in the silica tube as far from the platinum heating coil as possible. After the combustion chamber is evacuated, a controlled amount of air is admitted. A constant ratio of specimen weight to amount of air



With this equipment, the gaseous products, formed when organic materials are burned in air, can be readily collected for analysis. Amount of air, firing temperature, and size of specimen can be closely controlled to give a quantitative estimate of the amounts and kinds of combustion gases produced when such organic coatings as paints, asphalts, and plastic compounds are burned.

The principal combustion products of organic materials are carbon monoxide, carbon dioxide, and water. When organic materials contain other elements in addition to carbon, hydrogen, and oxygen, combustion yields other gases as well. Among these are ammonia from wool, cyanogen from silk, sulfur compounds from rubber, and chlorine compounds from chlorinated plastics. To generate, collect, and analyze combustion products, earlier investigators burned large, built-up specimens, including actual rooms and buildings. Subsequently, laboratory procedures were developed to replace such costly and time-consuming processes, but these methods were slow and of doubtful accuracy. The Bureau's recently developed method of burning organic materials provides a quick and comprehensive analysis of the gases as well as close control

is maintained by adjusting the specimen weight when combustion is initiated at higher temperatures.

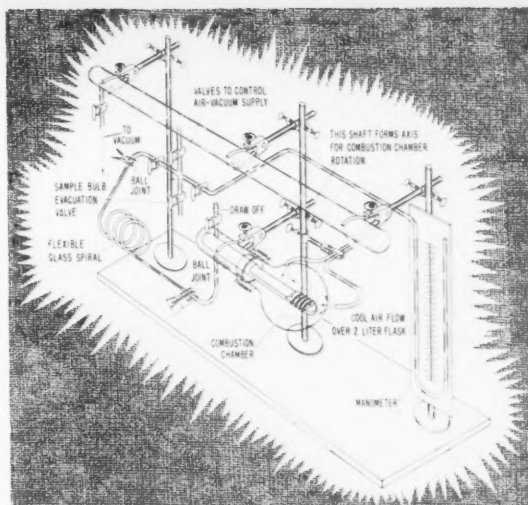
Current is applied to the platinum coil until the desired firing temperature is reached. The combustion chamber is inclined, the boat containing the specimen slides down the silica tube into the hot platinum coil, and the material begins burning. After the temperature has been held constant for the appropriate time, the current is turned off and the apparatus cools to room temperature. The combustion gases are now available for analysis in a mass spectrometer.

In a series of investigations performed with this equipment, the results showed that for every material examined the greatest variety of gases were produced at the highest initial firing temperature (550° C.). At this temperature, cracking and decomposition occurred

and small molecular fragments such as methane and hydrogen were formed from many of the specimens. Chlorinated plastics liberated chlorinated compounds at the higher temperatures, whereas at the lowest firing temperature the only chlorinated material produced was hydrogen chloride. In every case the amount of hydrogen chloride increased with the temperature.

Several plywood assemblies, both painted and unpainted, and a polyvinyl chloride coating were fired in quantities sufficient to consume all the oxygen in the combustion chamber if they had been left to burn entirely to carbon dioxide and water. These materials produced the largest concentration of combustion gases, and caused the greatest decrease in the oxygen concentration. This low oxygen concentration is typical not only of the closed system used here but also of large, open, ventilated systems such as burning rooms.

<sup>1</sup> For further technical details, see Method for the controlled burning of combustible materials and analyses of the combustion gases, by Alan Schriesheim, *J. Research NBS* **57**, 245 (1956) RP2715.



Apparatus for studying gaseous products resulting from the combustion of organic materials.

TABLE 1. Results obtained upon burning combustible materials in air

Temperature	Concentration of gases										
	Carbon dioxide	Carbon monoxide	Oxygen	Nitrogen	Methane	Methyl chloride	Ethylene	Propylene	1,3-Butadiene	Hydrogen chloride	Hydrogen
COATING—CHLORINATED HYDROCARBON A											
° C	%	%	%	%	%	%	%	%	%	%	%
250	0.9	0.1	17.7	74.0	---	---	---	---	---	2.4	---
400	11.1	.6	6.4	81.0	---	---	---	---	---	3.3	---
550	10.1	5.8	0.3	77.7	1.0	0.1	0.5	0.1	0.1	4.7	0.7
COATING—CHLORINATED HYDROCARBON B											
250	0.2	---	18.7	78.2	---	---	---	---	---	0.6	---
400	.8	---	21.4	76.2	---	---	---	---	---	.8	---
550	.4	---	17.0	81.4	---	---	---	---	---	1.1	---

## A HIGHLY ALKALINE pH STANDARD

TO increase the accuracy of pH measurements in the highly alkaline range, the Bureau is recommending a sixth pH standard<sup>1</sup> for use along with the five pH standards previously established. Consisting of a saturated solution of calcium hydroxide, the new standard will provide an additional fixed point in the standard pH scale which the Bureau maintains.

This scale was set up<sup>2</sup> in 1948 to meet the need for a single standard pH scale as a basis for accurate measurement of acidity and basicity in aqueous solutions. In the manufacture of many commercial products—for example, paper, textiles, dyes, and ceramics—the rapidity and efficiency of the processes depend upon the accuracy with which pH can be determined.

The NBS standard pH scale is defined in terms of several fixed points in much the same manner as is the International Temperature Scale. The primary standards of the pH scale are solutions whose pH values are only slightly affected by dilution or by accidental contamination with traces of acid or alkali from the walls of the container or from the atmosphere. The substances from which the standard solutions are prepared are, in turn, stable materials which may be obtained as certified standard samples from the Bureau. The five standards thus far established cover the pH range from 1.68 to 9.18 at 25° C. However, a highly alkaline standard has been needed for several years.

The new pH standard is a solution of calcium hydroxide saturated at 25° C. This reference solution is very easily prepared. No weighings are necessary, as a solution of reproducible composition can be made merely by shaking finely granular calcium hydroxide with water. The calcium hydroxide is prepared by igniting calcium carbonate conforming to American Chemical Society specifications for the reagent grade low in alkalis. The solid material must not be contaminated with soluble alkalis, but the presence of carbonate is of no concern, because calcium carbonate will precipitate from the solution at the time of preparation. The filtered solution of calcium hydroxide supersaturates readily and can normally be used over a wide range of temperatures without separation of the solid.

The pH of this solution is 12.45 at 25° C and is a rather sensitive function of temperature. Values of pH on the NBS conventional activity pH scale have been assigned at intervals of 5 degrees from 0° to 60° C. Although standard samples of calcium hydroxide are not yet available from the Bureau, highly pure preparations can be readily made from commercially available grades of calcium carbonate.

<sup>1</sup> For further details, see Calcium hydroxide as a highly alkaline pH standard, by R. G. Bates, V. E. Bower, and E. R. Smith, *J. Research NBS* **56**, 305 (1956) RP2680.

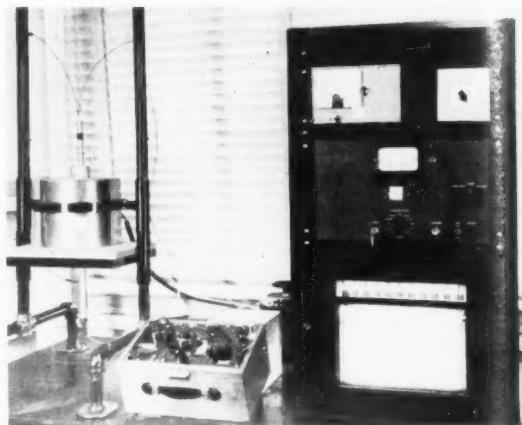
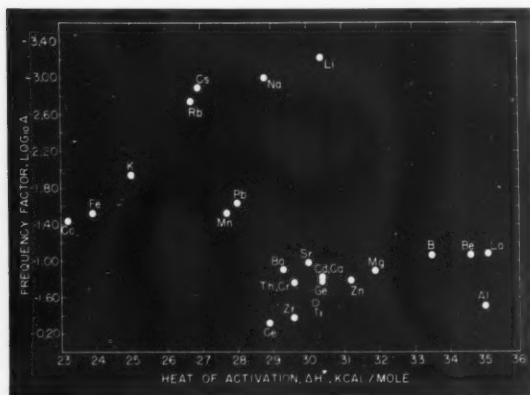
<sup>2</sup> Standardization of the pH scale, NBS Tech. News Bul. **31**, 138 (1947).

# CONTROLLING CONDUCTIVITY IN SILICATES

**A**N URGENT NEED for high-temperature electrical insulating materials has led to an investigation of methods for decreasing conduction in the basic glasses or frits used in vitreous ceramic coatings.<sup>1</sup> Results of the study indicate the general type of composition yielding coatings of high resistance. A better understanding of the basic mechanisms controlling electrical conduction in glasses has also resulted from the project, which was sponsored by the Office of Ordnance Research, Department of the Army. S. W. Strauss, D. G. Moore, W. N. Harrison, and L. E. Richards of the Bureau's enameled metals laboratory conducted the investigation. The research was aimed indirectly at producing ceramic-coated parts having "built-in" electrical resistance superior to that of mica.

Conductivity in vitreous silicates at high temperatures is generally thought to be the result of the net unidirectional migration of alkali ions through the interstitial holes of the silicate framework. For an ion to migrate, its free energy of activation must be exceeded. That is, sufficient energy must be supplied to break the chemical bond between the alkali ion and the oxygen, and to move the ion to the next interstitial position. Thus, the conduction process may be pictured as a series of migrations of conducting ions from interstice to interstice. To obtain a better insight into the conduction mechanism, the Bureau made a detailed study of the conductivity of simple glass-forming systems, including a large number of ternary lead silicates and lithium sodium silicates.

Specimens were prepared for test by spraying a thin layer of silver paint onto each face. Stainless steel electrodes, located within the heating core of a furnace, were placed in contact with the specimen and the furnace was heated at the rate of about 10 deg C per minute. Voltage from a 200-v dry cell was applied during the entire heating period, polarity being reversed at least once each 20 deg C, and current was measured at 10-deg C intervals. The resistances of the glasses were determined from the current, voltage, and dimensions of the specimens for temperatures up to 500° C in some cases.



Apparatus used to measure electrical resistivity of ceramic coating materials at elevated temperatures. Furnace, left, houses specimen in contact with two circular electrodes. The temperature of the top electrode, which contains an embedded thermocouple, is determined by a potentiometer, center. Second thermocouple, embedded in the furnace insulation, activates control unit at the upper left of the console. A recording potentiometer at the bottom of the console used a d-c preamplifier, immediately above, to graph the current passing through the specimen.

The observed variation in resistivity with change in composition indicates that conductivity depends on the nature of ions, being affected by such factors as their size and charge. Relationships between resistivity, composition, bonding energy, and experimental heat of activation of ternary lead silicates suggest that alkali ions may be the only conducting ions. Traces of alkali ions appear to decrease electrical resistivity, making purity of materials a main consideration in preparing alkali-free high-resistivity ceramic coatings. However, when the fusion point of the frit is very high, it may be necessary to add alkali ions to obtain workable coatings.

For lithium sodium silicates, a correlation was revealed between resistivity and composition, and between experimental heat of activation and composition. This finding indicates that substitution of two alkali ions for the same mole percent of either one leads to a more compact structure which decreases ionic migration. Thus, a composition containing approximately equimolar quantities of lithium oxide and sodium oxide has the most compact structure, and therefore the highest resistivity, in the ternary lithium sodium silicate system.

Graph showing the dependence of the number of charge carriers, indicated by the frequency factor, and the heat of activation on composition. Data was obtained by replacing 0.4 PbO in the base composition  $PbO:SiO_2$  with each of the ions listed. The number of carriers increases with the absolute value of the frequency factor,  $\log A$ .

The investigation leaves some questions unanswered, such as whether or not hydrogen ions from water in the glass contribute to conduction. However, the study represents a definite contribution to the conduction theory and provides data that should be helpful in the design of glasses, glazes, and ceramic coatings of specified resistivities.

<sup>1</sup> For further technical details, see Fundamental factors controlling electrical resistivity in vitreous ternary lead silicates, by S. W. Strauss, D. G. Moore, W. N. Harrison, and L. E. Richards, *J. Research NBS* **56**, 135 (1956) RP2658; Electrical resistivity of vitreous ternary lithium sodium silicates, by S. W. Strauss, *J. Research NBS* **56**, 183 (1956) RP2665; Effect of temperature on the electrical resistivity of several ceramic and silicone type coatings, by S. W. Strauss, D. G. Moore, and L. E. Richards, *ASTM Sp. Tech. Publ. No. 153*, 101-8 (1953).

## Postdoctoral Research Associates Appointed

FIVE young scientists who have shown special promise of becoming creative leaders in basic research have been awarded Postdoctoral Research Associateships for advanced study at the Bureau. The associateship program is sponsored jointly by the National Academy of Sciences, National Research Council and NBS.

The men, who have either received or shortly will be awarded their Ph. D. degrees, are scheduled to start work at NBS this year. They are: Earl C. Beaty, St. Louis, Mo.; Tucker Carrington, Aberdeen Proving Ground, Md.; Marvin Marcus, Vancouver, Canada; Robert A. Piccirelli, Washington, D. C.; and Wesley A. Robinson, Seattle, Washington.

The Research Associates will work under the direct supervision of scientists who are specialists in the associates' chosen fields. In addition to acquiring a wider basic knowledge, the Research Associates will have opportunities to work with and develop new scientific approaches and laboratory techniques. They also will be in close contact with other NBS scientists and the national and international scientific leaders with whom the Bureau is associated.

Postdoctoral study and research lasting for a year or more is a tradition among scientists. Many of the country's scientific leaders used such work to round out their education before settling on the field of research for which they are best known. In recent years the number of opportunities for such postdoctoral appointments has not kept up with the need; in fact, one of the most sought after fellowships in the physical sciences in existence since World War I has been discontinued.

Recognizing this need, the National Academy of Sciences and the Bureau worked out the research associateship program to provide advanced training for promising young American scientists. The associateships were first announced in 1954. This year some 20 areas of study were made available to the applicants for the associateships.

They were: Applied mathematical statistics, numerical analysis, pure and applied mathematics, experimental atomic and nuclear physics, experimental thermodynamics, fundamental problems in physical measurement and computer design, interaction of radiation with matter, low temperature physics, mechanics of fluids and solids, molecular structure and spectroscopy,

radiological physics, radio wave propagation, solid state physics, statistical mechanics, visual psychophysics, physics and chemistry of polymers, and analytical, inorganic, organic, and physical chemistry.

The Bureau is vitally concerned with the expanding frontiers of science, particularly in basic research on physical standards and constants, the properties of matter, and measurements. Its diverse program offers interesting opportunities for postdoctoral fellowship study. The research associateship program is directed by Wallace R. Brode, Joseph Hilsenrath, and David E. Mann of NBS. Supervising the program for the National Research Council are M. H. Trytten, Director of Scientific Personnel, and Claude Lapp, Director of the Fellowship Office.

In applying for the postdoctoral positions, all applicants sought close association with particular staff members. Also, several applicants expressed interest in using some of the Bureau's unusual equipment such as the special solenoid for the very precise measurement of electrical quantities and the versatile SEAC digital computer.

The five scientists chosen for study during 1956-57 and their research fields are as follows:

EARL C. BEATY, Washington University (Ph. D.). Dr. Beaty plans to work on the measurement of positive ions in gases. His advisor will be Lewis M. Branscomb, Chief of the Atomic Physics Section.

TUCKER CARRINGTON, California Institute of Technology (Ph. D.). Dr. Carrington intends to study energy transfer processes involving OH molecules in flames. He will be working under supervision of H. P. Broida, of the Temperature Physics Section.

MARVIN MARCUS, University of California (Ph. D.). Dr. Marcus plans to study the eigenvalue inequalities for finite matrices. Olga Taussky-Todd of the Applied Mathematics Division will be his supervisor.

ROBERT A. PICCIRELLI, Catholic University of America (Ph. D.). Dr. Piccirelli's field of study will center around the application of statistical mechanical methods to systems containing charged particles. He will have as supervisors C. W. Beckett and M. S. Green of the Thermodynamics Section.

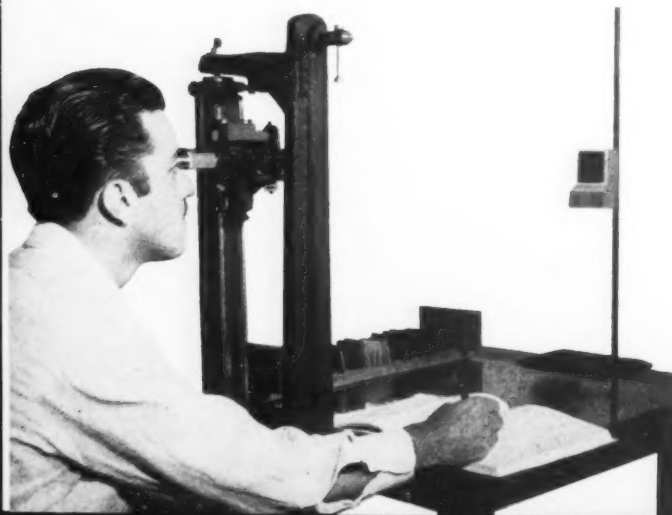
WESLEY A. ROBINSON, University of Washington (Ph. D.). Dr. Robinson plans to study sound absorption in nuclear reorientation. His advisor will be Martin Greenspan of the Sound Section.

# LEATHER

## RESEARCH

**I**N MANY RESPECTS leather is a unique material, possessing a number of desirable physical properties, such as tearing strength, flexibility, and porosity, that make it particularly well suited to use in shoes, gloves, and other articles of clothing. For specific applications, these properties can be controlled to a considerable extent by proper selection of skins, tanning agents, and finishing processes. A knowledge of the structure of collagen, the basic leather forming protein of hides, as well as an understanding of the mechanism of tanning, is thus of direct importance to tanners, shoe manufacturers, and other industrial users of leather.

To provide basic information of this kind for Government and industry, the National Bureau of Standards has, over the past 40 years, carried on an extensive program of research and development in the field of leather.<sup>1</sup> This program, now under the direction of J. R. Kanagy, has included fundamental studies of collagen, development of concepts for the mechanism of tanning, and measurement of the physical and chemical properties of collagen and leather. Much of the information thus obtained has been applied to the improvement of leather products and the development of standard methods for their evaluation.



### Fundamental Physicochemical Studies

Because of the complex nature of collagen, a large proportion of the Bureau's leather investigations have combined both physical and chemical studies. An example is a series of investigations of the interactions of leather and collagen with water in various forms. Leather excels in its ability to transfer water vapor from a region of high humidity to one of lower humidity. Because of the relation of this property to shoe comfort and the removal of perspiration from shoes, water-vapor transfer through leather has been extensively studied at NBS.<sup>2</sup> The strong adsorptive capacity of leather and collagen for water and water vapor also has an important effect on the properties of leather. This has led to a series of studies on water adsorption and its variation with temperature, tannage, and other factors. These studies are being extended to include heats of wetting of leather and other fibrous polymers.

The physical constants of leather fibers are perhaps of less immediate practical importance. Nevertheless, they are of great value in understanding the complex nature of collagen and the processes involved in converting collagen to leather. In general, studies of fiber constants are made under varying conditions of tannage, moisture content, temperature, composition, and other factors, so that extensive data result.

To learn more about the thermodynamic properties of leather, data have been obtained on its density, compressibility, expansivity, and specific heat. In the course of this work, investigators encountered interesting phenomena which are being explored further. For example, the expansivity measurements led to studies of shrinkage rates, with resulting data on heats and entropies of activation. Likewise, compressibility measurements at the high pressures required for solid materials led to similar studies on synthetic polymers.

In measuring leather spread, telescope comparator is used to determine the increase in length of a sample after pressure has been applied along the smallest dimension.

NBS Technical News Bulletin

Data on specific heats are now being accumulated in connection with basic studies on interactions of moisture and leather.

## Basic Chemistry of Collagen

Skin collagen, being a natural fibrous protein, is a highly complex polymer that occludes extraneous materials, such as fats, salts, and other proteins. Thus, fundamental chemical studies of collagen first required investigation of methods for removing extraneous materials so that a chemically reproducible purified collagen might be prepared.

The chemistry of the purified collagen that was finally obtained has been intensively studied, providing data that should be of great value in determining its structure and the mechanism of tanning. Besides studying relatively simple chemical reactions, such as esterification, deamination, and various tanning procedures, the Bureau has determined the combining weights and the reactive basic groups. It has also studied the amino acid structure and the amide nitrogen content of collagen.

Recently, a rapid chromatographic method was developed for determining the amino acid content of collagen.<sup>3</sup> This quantitative technique can be applied not only to collagen itself but also to its derivatives and degradation products. In contrast to other methods currently employed, it enables the analyst to follow with a high degree of detail the reactions and changes in which collagen may be involved. It is thus expected to provide a useful tool that will aid in further understanding the structure of collagen and the properties of leather.

## Physical Properties

The behavior of leather in service is largely determined by the physical properties of the leather matrix. Mechanical properties that have been investigated extensively include tensile strength, stretch, tearing strength, stiffness, bursting strength, rigidity, and flexural resistance. Investigations of thermal properties have involved shrinkage temperature, area stability, and thermal conductivity. Other studies have dealt with electrical resistance, dielectric constant, and structural properties such as pore-size distribution in the fibers and leather.

Recently, fundamental information regarding the structure of leather and collagen fibers was obtained from studies of pores in leather.<sup>4</sup> Pressure porosimeter and electron microscope studies revealed the presence of large numbers of extremely small pores—less than a millionth of an inch in radius—in both leather and collagen. Quantitative information was obtained on pore-size distribution within the individual collagen fibrils. These data should aid the leather technologist in understanding the swelling and shrinkage accompanying water-leather interactions. They are also expected to shed additional light on the ability of leather to transmit or absorb water vapor, tannins,



Steam distillation is used to separate and identify fungicides. This work is basic to the development of Federal Specifications for leather fungicides.

or impregnants. The permeability of leather to water vapor, which makes it so well suited to use in footwear, is undoubtedly related to the presence of pores.

## Leather Technology

In 1924, a survey by the U. S. Department of Commerce stated that 40 percent of the vegetable tanning materials consumed in the United States in 1922 were imported. It also pointed out that 99 percent of the chrome ore used in the United States at that time came from abroad. In view of the obvious importance of a substitute tanning material to the national economy, the Bureau initiated research on synthetic tanning agents.

Representative syntans of various types were prepared and were evaluated by tanning tests. The published results helped lay the foundation for the development of present-day satisfactory syntans by industry. An investigation of the tanning properties of sulfite cellulose, a by-product of the paper industry, demonstrated that this material could be used in conjunction with vegetable tanning extracts. Later, during World War II, it was shown that iron could be substituted for chromium during an emergency.



During World War II military and civilian shoe requirements were so great that the tanning industry had difficulty in expanding operations sufficiently to meet the increased demand. Of chief concern were the acute shortages of hides and tannins. Recognizing the need for conservation, the Army Quartermaster Corps sponsored research at the Bureau to improve the stability and serviceability of leather and to develop test methods for military items. The Bureau investigated several oil and wax treatments, which



#### Quantitative determination of amino nitrogen in leather extracts or hydrolysates.

were adopted by the Armed Forces to increase the wear resistance of sole leather.

After the war, research directed toward improving the serviceability of leather was continued. This work aimed not only to increase the wear resistance of sole leather but to improve low-grade hide areas so that more first-quality soles might be cut from the same hide. Ultimately both these objectives were attained by development of a method for impregnating leather with polymers.

The tightly woven condition of the natural fibers of leather tends to limit the materials with which it may be impregnated. Thus, the Bureau's early attempts to use various solutions of commercial polymers as impregnants were not successful, and research was for a time directed toward impregnating the leather with monomers for polymerization *in situ*. Then, as a result of other research on pore-size distribution in leather and particle-size distribution in rubber latex, it became apparent that leather could be impregnated by soaking the crust leather (tanned, but not finished) in solutions of specially prepared or selected commercial polymers. On the basis of these findings, a method of treatment was developed.

Laboratory tests have shown that the treatment increases wear by about 80 percent and reduces water absorption by about half. Because crust sole leather containing a minimum amount of uncombined tannins is used, considerable saving in tanning materials is effected. Furthermore, impregnation may increase the wear of belly and shoulder leather to equal that of high-quality sole leather.

To bridge the gap between laboratory investigation and large-scale industrial application of the process, pilot-plant studies are being conducted at NBS under sponsorship of the Navy Bureau of Supplies and Accounts. These studies have shown that by varying the impregnating process, long wearing leathers can be produced having various degrees of waterproofness, flexibility, and water-vapor transmission. Methods have also been developed for imparting a finished appearance to the impregnated leather.

### Stability and Serviceability

The aging qualities of leather depend principally on its tannage, environment, and use. However, investigations of the mechanism of degradation have shown that highly ionizable acids, either added during manufacture or adsorbed from the atmosphere, are a major factor in leather deterioration.

Over 20 years ago the Bureau made a thorough study of the effect of acid on leather and published a

In study of leather fungicides, the treated specimens of leather are kept in an oven at 30° C to stimulate a tropical environment. A small amount of water in the bottom of each specimen jar provides the necessary humidity.

To measure the water vapor permeability of leather, a sample, cut in the form of a circle, is placed across the opening of a shallow cup containing a drying agent. The cup and contents are first weighed in the balance and then hung in a humidity chamber (lower left), where the moisture that passes through the leather sample is absorbed by the drying agent. A second weighing then determines the amount of absorbed moisture, a measure of the water-vapor permeability of the sample.

series of papers on the subject. Some of the more important results of this investigation were the development of a standard procedure for determining acidity in leather and the determination of the optimum pH to prevent change in the properties of leather during prolonged storage. Other factors in leather deterioration that the Bureau has studied include temperature, oxygen, moisture, and the catalytic effect of traces of copper and iron salts in the leather.

Before World War II mildew on leather was not considered very important and little effort was made to prevent its growth. However, military forces stationed in tropical areas soon found that the growth of mildew on numerous items of equipment was a serious problem. The Army therefore initiated a project at the Bureau for the development of fungicidal treatments and test methods.<sup>6</sup>

These studies showed that the principal effect of mildew on leather, other than appearance, is the removal of greases, which causes stiffness and loss in strength. As a result of the investigation, a specification for the fungicidal treatment of leather was prepared and quantitative methods were developed for the determination of fungicides in leather. This work has been continued with the development of new fungicides and establishment of procedures for reliable evaluation of all fungicidal materials.

## Test Methods

Much of the Bureau's work on leather has been concerned with the development of new or improved methods or equipment for testing leather and leather products. The results have been used both in procurement specifications and in evaluation and development work.

For many years the accurate determination of moisture in leather was a serious problem to analysts, as the results for all other chemical constituents are expressed on the oven-dry basis. In 1941 NBS research

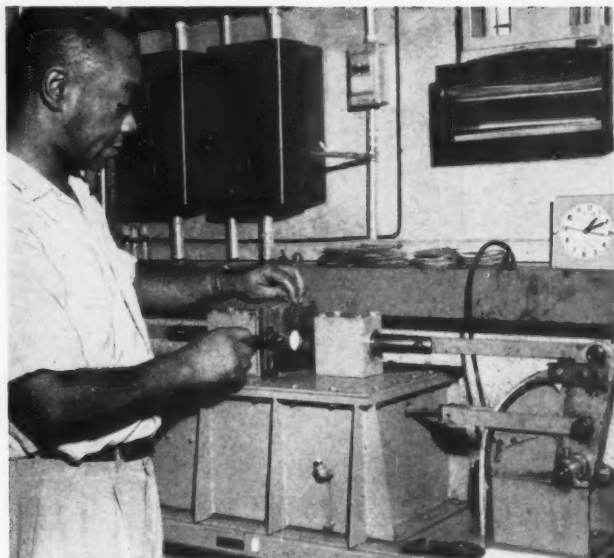
High-pressure equipment used for compressibility experiments. Designed primarily for study of leather shrinkage, this apparatus has been found of value for studying the high-pressure properties of a number of other high polymers. Mounted in the press is a thick-walled cylindrical steel bomb which contains the polymeric specimen immersed in a pressure-transmitting liquid. Pressure is applied to force a tight-fitting piston into the bomb from above, increasing the hydrostatic pressure within and compressing the specimen. The dial gage mounted on the ram that drives the piston into the bomb shows depth of penetration.



showed that control of the atmospheric humidity in the drying oven would permit moisture determinations to be made with considerable precision. This method has since been adopted as the ultimate standard in Federal Specifications.

Because the properties of leather vary considerably over the area of a hide, it is important that a test sample represent as nearly as possible the average for the hide. Bureau scientists devised a sampling procedure<sup>7</sup> for shoe upper leather that permits selection of sampling





**Right:** Chromatograms used in determining the amino acid content of collagen, parent substance of leather. Each component appears as a separate colored patch on a sheet of cellulose paper. The amount of each amino acid present is determined by comparing optical densities of the extracted patches with appropriate calibration curves. **Left:** Finding the impact resistance of a leather sample. Horizontal rods alternately punch the sample (circular opening, center) in opposite directions until failure occurs.

locations so that the fewest specimens are required to give the desired accuracy for a particular property. The sampling procedure is based on a mathematical relationship involving the coefficient of correlation between the value obtained for a particular test from a specific location and the average for that test over the corresponding side. The most suitable sampling location on a hide for all tests required in acceptance testing was also determined.

A number of techniques and devices have been worked out for making performance tests of leather products. Much of this work has dealt with water-vapor permeability. Other developments have been concerned with measurement of abrasion resistance, flex life, accelerated aging, and compressibility.

Recently, in work sponsored by the Office of the Quartermaster General, the Bureau has developed a non-destructive method for testing leather, based on the transmission of sound waves.<sup>6</sup> The chief instrument employed is a pulse propagation meter which measures and records the speed of a generated sound pulse through the leather. As a result, the specimen under test is left unharmed, in contrast to the tearing or other destructive effects of other test procedures. Good correlation has been found between sonic measurements and the results of tensile and breaking elongation tests.

### Future Plans

In the near future the Bureau expects to begin a study of leather degradation by radiation. The newly de-

veloped technique for amino acid determination will be used in this work. This method of approach should show the exact point of attack in the collagen molecule and thus give a clearer explanation of the mechanism of breakdown.

Recently developed methods for determining particle size of polymers will also be utilized to study the size of particles in tannins and leather impregnants. Comparison of these results with data on leather pore size should not only provide additional information on the mechanism of tanning but should also make possible a better understanding of the nature of the tanned material.

<sup>1</sup> For further details and a bibliography of NBS publications in the leather field, see Leather research and technology at the National Bureau of Standards, by Everett L. Wallace NBS Circular 560 (1955); available from the Superintendent of Documents, U. S. Government Printing Office, 15 cents.

<sup>2</sup> Water vapor permeability of leather, NBS Tech. News Bul. **34**, 163 (Nov. 1950).

<sup>3</sup> A rapid quantitative analysis of collagen, NBS Tech. News Bul. **40**, 65 (May 1956).

<sup>4</sup> Micropores in leather, NBS Tech. News Bul. **39**, 68 (May 1955).

<sup>5</sup> Improved techniques for impregnating leather, NBS Tech. News Bul. **39**, 96 (July 1955).

<sup>6</sup> Prevention of mildew on leather, NBS Tech. News Bul. **32**, 81 (July 1948).

<sup>7</sup> Sampling of upper leather for shoes, NBS Tech. News Bul. **35**, 6 (Jan. 1951).

<sup>8</sup> A sonic technique for testing leather, NBS Tech. News Bul. **40**, 35 (March 1956).

# CRYOGENIC ENGINEERING CONFERENCE

**M**ORE than 400 scientists and engineers attended the 1956 Cryogenic Engineering Conference held September 5-7 at the NBS Boulder (Colo.) Laboratories. The second meeting of this kind to be held in the United States, the Conference dealt with problems of engineering research and development at temperatures below 150° K (-190° F). In this temperature range the behavior of materials is markedly different from that observed at ordinary temperatures.

R. B. Scott, Chief of the NBS Cryogenic Engineering Laboratory, called the Conference to order on Wednesday morning, September 5. Mr. Scott then introduced Dr. F. W. Brown, Director of the Boulder Laboratories, who briefly welcomed the delegates. The remainder of the Conference was devoted to technical papers and discussion on 50 topics of current interest in the fields of cryogenic processes, cryogenic equipment, cryogenic properties, cryogenic applications, and bubble chambers. On Thursday evening the delegates attended a banquet at which the invited speaker was Dr. E. U. Condon, Head, Department of Physics, Washington University.

Considerable interest was shown in the group of papers dealing with liquid-hydrogen and liquid-helium bubble chambers for detecting and observing the behavior of high-energy particles. The 10-inch bubble chamber now operating at the University of California Radiation Laboratory was described by R. L. Blumberg, J. D. Gow, and A. J. Schwemin of the Radiation Laboratory. In this apparatus high-energy particles are passed through the superheated liquid at speeds approximating that of light. From a high-speed photographic record of the tracks of the resulting bubbles, studies are made of the interaction of the atomic particles with matter. The bubble chamber fits into a magnet capable of providing a 12,000-gauss magnetic field, which deflects charged particles traversing the chamber so that investigators now may determine particle momentum.

A larger bubble chamber is now being designed at the University of California Radiation Laboratory with the assistance of the National Bureau of Standards. This apparatus was described by D. B. Chelton and D. B. Mann of NBS, and R. A. Byrns of the Radiation Laboratory. The larger chamber will allow more extensive observations to be made of atomic particle interactions. It will contain approximately 500 liters of liquid hydrogen, will have a 23- by 75-inch glass window weighing several hundred pounds, and will be maintained at the desired operating condition by a closed-cycle hydrogen refrigerator of approximately 2,000 watts.

R. H. Kropschot and R. P. Mikesell of NBS reported on their experiments with glass at low temperatures. These investigators measured the strength and fatigue properties of BSC-2 optical glass at various temperatures within the range from 20° to 296° K. Their data will be of value in constructing the window of optical glass needed for the large bubble chamber.

An ultrapowerful magnet constructed at Los Alamos Scientific Laboratory was discussed by Dr. H. L. Laquer of the University of California. Ultimately it is hoped that this magnet can be used to cool matter to temperatures lower than any previously attained. The magnet differs from most electromagnets in that it requires only  $1/100$  of the electric power needed to operate a room temperature magnet of equal size. When cooled to liquid hydrogen temperatures, the magnet coils become excellent electrical conductors.

A new practical application for liquid hydrogen was described by M. D. Andonian of the Cambridge Corporation, who told how engineers doing work for the Air Force Cambridge Research Center have found a way to replenish gas lost from a high-altitude weather balloon while it is still in flight. A specialized storage container has been developed that holds nearly 120 gallons of liquid hydrogen and can be attached to the balloon for automatic refueling. With this device, weather balloons will now be able to remain aloft many hours longer and make more extensive observations.

A potentially low-cost method for obtaining heavy hydrogen by low-temperature distillation of liquid hydrogen was discussed in a paper by T. M. Flynn, D. H. Weitzel, K. D. Timmerhaus, P. C. Vander Arend, and J. W. Draper of NBS. With the advent of electrical energy from nuclear power, a considerably increased demand for heavy water can be expected, and its cost will be a significant factor. The heavy hydrogen obtained by the distillation process can then be used in making heavy water. The NBS Cryogenic Engineering Laboratory is now constructing a pilot plant to evaluate the process on a larger scale.

G. F. Tanza of Garrett Corporation reported on a ball bearing that will operate without any lubrication at temperatures very close to absolute zero and at speeds as great as 10,000 rpm. The ball bearing is of stainless steel and utilizes a plastic retainer. It will operate continuously for at least 150 hours at temperatures ranging from -420° to +100° F.

An invited paper on gas refrigeration machines was given by Dr. J. W. L. Kohler of the Philips Research Laboratories, Eindhoven, Netherlands. He described an unusually compact, single-cylinder water-cooled apparatus for gas liquefaction. This machine is unique in that the gas does not pass through the working parts, but condenses on the surface of the cylinder head. Thus, an exceptionally pure product is obtained, free from lubricating oil or similar contamination. Designed primarily for liquid air production, this apparatus can, with simple modification, be used for liquefying other industrial gases. Production begins in as little as 15 minutes after starting the operation, whereas conventional methods require more than 2 hours before the processes begin.

Dr. S. C. Collins of the Massachusetts Institute of Technology described experiments he is conducting to liquefy helium and hydrogen at a cost only slightly greater than that of liquefying air. A heavy-duty ma-

chine that has been built at MIT for this purpose will run for years with only a nominal maintenance cost. This machine is now experimentally producing liquid helium for as little as \$4.00 a quart. Conventional methods usually result in helium liquefaction costs of, at least, \$25.00 a quart. Served by a three-stage 90-horsepower helium compressor, the apparatus has a capacity of about 45 liters of liquid helium per hour or about 50 liters of liquid hydrogen.

Development of a new catalyst for converting ordinary liquid hydrogen to parahydrogen was discussed

in a paper by D. H. Weitzel, O. E. Park, J. W. Draper, and K. D. Timmerhaus of NBS. This new catalyst, an iron hydroxide gel, is nearly 50 times more effective than the catalysts previously used for this purpose. In the paraform liquid hydrogen can be stored for long periods with greatly reduced loss. V. J. Johnson of NBS described a method for installing the new catalyst in the Bureau's large liquefiers to increase their rate of production as much as 18 percent.

Present plans call for publication of the Conference proceedings early in 1957.

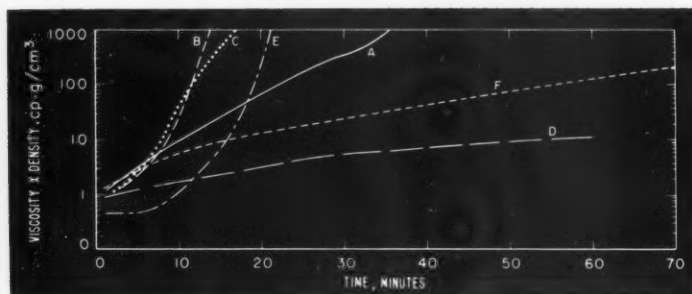
## Improvement of Plastic Dental Resins

**W**ITH PROPER SHADING, the plastic, polymethyl methacrylate, can be made into dental fillings that have excellent appearance. Generally, such fillings are made from monomer-polymer mixtures that are designed to harden at room temperature. Sometimes, however, the mixtures do not harden sufficiently or quickly enough. To improve the quality of the hardened product, a study was made of a number of factors that may affect the polymerization mechanism. Partial results of the investigation have recently been made available.

Data regarding the effect of a single accelerator on polymerization at room temperature have been compiled by H. J. Caul and G. C. Paffenbarger, American Dental Association research associates at the Bureau, and W. T. Sweeney of the Bureau's dental research

of radicals and thus speed polymerization. In practice, the dentist uses an accelerator dissolved in the liquid monomer and a polymer powder that contains benzoyl peroxide initiator. The liquid is added to the powder and the mixture placed in the cavity before polymerization is completed.

Values for residual monomer, peak temperatures, and physical properties of the hardened plastic were determined on samples of resin prepared from one polymer with monomer containing different proportions of the same accelerator, dimethyl-*p*-toluidine. It was found that up to a certain amount, larger proportions of accelerator give faster setting times, less residual monomer in the finished product, and improved physical properties, such as indentation and yield strength.



**Polymerization of methyl methacrylate in the presence of *p*-toluene-sulfinic acid-amine salts at 37° C and 2 percent benzoyl peroxide concentration.** A, aniline salt, 1 percent; B, methylamine salt, 1 percent; C, dimethyl-*p*-toluidine salt, 2 percent; D, di-*n*-butylamine salt, 2 percent; E, di(β-hydroxyethyl)-*m*-toluidine, 1 percent; F, *p*-toluene-sulfinic acid, 2 percent.

laboratory. They found that the residual monomer content is directly related to the percent of accelerator used.<sup>1</sup> Further investigations with respect to different kinds of accelerators were conducted by G. M. Brauer, F. R. Burns, R. M. Davenport, and W. C. Hansen of the Bureau staff.<sup>2,3</sup> The data on setting time and physical properties should be of value in formulating improved dental resins.

Methyl methacrylate is liquid at room temperatures. In order to produce the rigid plastic, the monomer is generally subjected to elevated temperatures, with benzoyl peroxide added to initiate polymerization. When heated, benzoyl peroxide decomposes to form free radicals that link up with the monomer to initiate chain growth. However, benzoyl peroxide does not form enough free radicals at room temperature to start polymerization. Therefore, amine accelerators have been added to dental resins to promote the formation

Although they improve physical properties, most accelerators undergo side reactions and cause the resin to become highly colored. The resin manufacturer controls color by adding pigments, but the resulting shade has sometimes been unstable and has changed after the restoration is in place. Other accelerators produce a fast setting, but the finished product is not strong enough. Looking for an accelerator that would form a strong, colorless product, the Bureau studied the effect of nearly 100 accelerators on the resin setting time, physical properties, and visual appearance. Setting times of monomer-polymer mixtures were determined by means of a penetration test. Polymerization of monomer solutions was followed with an ultrasonic viscometer.

The Bureau found that only a few tertiary aromatic amines are efficient polymerization accelerators. Many amines cause a fast initial reaction, but the polymeriza-

tion does not go to completion. The relative setting times of monomers did not always correlate with those of monomer-polymer mixtures although the same amine accelerators were used. In general, it was found that such physical properties as resistance to indentation and recovery after indentation are improved with shorter setting time. Resins polymerized in the presence of dimethyl-*p*-toluidine and 2,2'-(*m*-tolylimino)-diethanol show the most desirable physical properties. However, the polymeric products formed in the presence of all the efficient amines are colored.

Attempting to find other accelerators that would not impart color to the product, the Bureau synthesized a number of sulfinic acid derivatives. It was known that *p*-toluidine sulfinic acid dissolved in the monomer would initiate polymerization without any induction period. However, this acid is unstable and readily oxidizes to form a sulfonic acid that does not activate polymerization. A number of aromatic amine salts of this sulfinic acid were synthesized and studied as poly-

merization agents. Such compounds as the methylaniline or dimethyl-*p*-toluidine salt of *p*-toluene sulfinic acid were found to be excellent accelerators and to have much better storage stability than the free acid. However, no marked improvement in the color of the products was obtained. Aliphatic amine salts or the free acid produce colorless products, but they are less effective accelerators.

<sup>1</sup>For further technical details, see Relationship between residual monomer and some properties of self-curing dental resins, by H. J. Caul, W. T. Sweeney, and G. C. Paffenbarger, *J. Am. Dental Assoc.* **53**, 60 (1956).

<sup>2</sup>For further technical details, see Sulfinic acid derivatives as accelerators in the polymerization of methyl methacrylate, by G. M. Brauer and F. R. Burns, *J. Polymer Sci.* **19**, 311 (1956).

<sup>3</sup>For further technical details, see Accelerating effect of amines in the polymerization of methyl methacrylate, G. M. Brauer, R. M. Davenport, and W. C. Hansen, *Modern Plastics* **34**, 153 (1956).

## Dr. Frederick Seitz Appointed to Visiting Committee

DR. FREDERICK SEITZ, one of the Nation's leading authorities on solid state physics, has been appointed to the National Bureau of Standards Statutory Visiting Committee. Dr. Seitz is a Professor of Physics at the University of Illinois and Chairman of the Governing Board of the American Institute of Physics.

The Visiting Committee, which is authorized by the Bureau Enabling Act of 1901, consists of five prominent scientific and industrial leaders appointed by the Secretary of Commerce. Their function is to visit the Bureau at least once a year and report to the Secretary "upon the efficiency of its scientific work and the condition of its equipment."

Appointed for a 5-year period, Dr. Seitz replaces Dean J. H. Van Vleck of Harvard University whose term expired this year. The other members of the Visiting Committee (with dates of expiration of ap-

pointment) are Dr. M. J. Kelly, President of Bell Telephone Laboratories, Inc., (1957); Dr. Clyde E. Williams, President of Battelle Memorial Institute, (1958); Crawford H. Greenewalt, President of E. I. duPont de Nemours & Company, (1959); and Dr. D. W. Bronk, President of the National Academy of Sciences, (1960).

Dr. Seitz was born in San Francisco, California, in 1911. He received his A. B. from Stanford University in 1932 and his Ph.D. in physics from Princeton University in 1934. Prior to joining the Physics Department at the University of Illinois, he was associated with Rochester University, General Electric Company, the University of Pennsylvania, and Carnegie Institute of Technology.

He is a fellow of the Physical Society and a member of the National Academy of Sciences, the Philosophical Society, and the Institution of Mining and Metallurgy.

## Metal Physics Section Established

A NEW SECTION for fundamental investigations of metal systems has been established at the Bureau. Designated the Metal Physics Section, the new group will be a part of the Metallurgy Division, and will supplement the present program of the other four sections of the Division. These sections are concerned with the broad fields of mechanical, chemical, and thermal metallurgy, and corrosion.

In general, the NBS metallurgy program is directed toward a better understanding of the properties and behavior of metals in order that new or improved metals and alloys may be developed for better performance in established uses and to meet the requirements of new applications. Such a program requires accurate knowledge of the intimate structure of metals

and alloys, and of the changes in structure that result from fabrication operations, heat treatment, and applied stress.

Primary purpose of the Metal Physics Section will be to further this knowledge through experimental and theoretical investigations of both single metal and alloy systems. The ultimate objective of this work will be the attainment of a clearer concept of these systems in terms of the characteristics of the constituent atoms and their arrangement in metal crystals. Specific areas in which research will be conducted include diffusion in metals, electric and magnetic properties, interstitial compounds, and solid state transformations.

The new section will be headed by Lawrence M. Kushner, formerly of the Surface Chemistry Section.

UNITED STATES  
GOVERNMENT PRINTING OFFICE

DIVISION OF PUBLIC DOCUMENTS  
WASHINGTON 25, D. C.

OFFICIAL BUSINESS

PENALTY FOR PRIVATE USE TO AVOID  
PAYMENT OF POSTAGE, \$300  
(GPO)



TECHNICAL  
NEWS  
BULLETIN

U. S. DEPARTMENT OF COMMERCE  
SINCLAIR WEEKS, *Secretary*  
NATIONAL BUREAU OF STANDARDS  
A. V. ASTIN, *Director*

November 1956 Issued Monthly Vol. 40, No. 11

For sale by the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. Subscription price, domestic \$1.00 a year; 35 cents additional for foreign mailing; single copy, 10 cents. Use of funds for printing this publication approved by the Director of the Bureau of the Budget (March 29, 1956).

Contents

	Page
Motion pictures of metal fatigue.....	153
Apparatus for etching fine points.....	155
Controlled burning of combustible materials.....	156
A highly alkaline pH standard.....	157
Controlling conductivity in silicates.....	158
Postdoctoral Research Associates appointed.....	159
Leather research.....	160
Cryogenic Engineering Conference.....	165
Improvement of plastic dental resins.....	166
Dr. Frederick Seitz appointed to Visiting Committee.....	167
Metal Physics Section established.....	167
Publications of the National Bureau of Standards.....	168

Publications of the National Bureau of Standards

Journal of Research of the National Bureau of Standards, volume 57, No. 4, October 1956 (RP2708 to RP2715 incl.). 60 cents. Annual Subscription \$4.00.  
Technical News Bulletin, volume 40, No. 10, October 1956. 10 cents. Annual subscription \$1.00.  
Basic Radio Propagation Predictions for February 1957. Three months in advance. CRPL 146. Issued October 1956. 10 cents. Annual subscription \$1.00.  
Journal of Research, volume 57, No. 4, October 1956. 60 cents. Single copies of Research Papers appearing in the Journal are not available for sale. The Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., will reprint 100 or more copies of a Research Paper. Request for the purchase price should be mailed promptly to that office.

Research Papers

RP2708. Infrared spectra of D-talose monobenzoate and related substances. H. S. Isbell, J. E. Stewart, H. L. Frush, J. D. Moyer, and F. A. Smith.  
RP2709. Small oil-free bearings. Hobart S. White.  
RP2710. Preliminary spectroradiometric measurements of the solar constant. Ralph Stair and Russell G. Johnston.  
RP2711. Infrared spectrum of acetylene. Harry C. Allen, Jr., Eugene D. Tidwell, and Earle K. Plyler.  
RP2712. Variation of peak temperature with heating rate in differential thermal analysis. Homer E. Kissinger.  
RP2713. Mass spectrum of sulfur vapor. Paul Bradt, Fred L. Mohler, and Vernon H. Dibeler.  
RP2714. Calibration of vibration pickups by the reciprocity method. Samuel Levy and Raymond R. Bouche.  
RP2715. Method for the controlled burning of combustible materials and analyses of the combustion gases. Alan Schriesheim.

Circulars

C578. Suggested practices for electrical standardizing laboratories. Francis B. Silsbee. 15 cents.

Building Materials and Structures Reports

BMS147. Effects of mineral additives on the durability of coating-grade roofing asphalts. Sidney H. Greenfeld. 20 cents.

Publications in Other Journals

Electrical conduction in magnesium stannide at low temperatures. H. P. R. Frederikse, W. R. Hosler, and D. E. Roberts. Phys. Rev. (American Inst. of Physics, Inc., 57 E. 55th St., New York 22, N. Y.) 101, No. 6, 1653-1660 (March 15, 1956).  
Effect of 20-year marine atmosphere exposure on some aluminum alloys. Fred M. Reinhart and George A. Ellinger. Symposium on Atmospheric Corrosion of Non-Ferrous Metals. (American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.) 175, 47-66 (1955).  
Processing denture base resins: heat-curing type. John W. Stanford and George C. Paffenbarger. J. Am. Dental Assoc. (222 E. Superior St., Chicago 11, Ill.) 53, 72-73 (July 1956).  
VHF propagation measurements in the Rocky Mountain region. R. S. Kirby, H. T. Dougherty, and P. L. McQuate. Trans. IRE (Inst. of Radio Engineers, Inc., 1 E. 79th St., New York 21, N. Y.) PGVC-6, 13-19 (July, 1956).  
Oblique-incidence measurements of the heights at which ionospheric scattering of the VHF radio waves occurs. V. C. Pineo. J. Geophys. Research (The Johns Hopkins Press, Baltimore 18, Md.) 61, No. 2, 165-169 (June, 1956).  
A photometric unit for the airglow and aurora. J. Atm. Terrest. Phys. (Academic Press Inc., 125 E. 23d St., New York 10, N. Y.) 8, 345-346 (June, 1956).  
Shielding of a transient electromagnetic dipole field by a conductive sheet. J. R. Wait. Can. J. Phys. (National Research Council, Ottawa, Canada) 34, 890-893 (August, 1956).  
On the waveform of a radio atmospheric at short ranges. J. R. Wait. Proc. IRE (Inst. of Radio Engineers, Inc., 1 E. 79th St., New York 21, N. Y.) 44, 1052 (August, 1956).

Publications for which a price is indicated are available only from the Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C. (foreign postage, one-third additional). Reprints from outside journals are not available from the National Bureau of Standards but can often be obtained from the publishers.

53  
55  
56  
57  
58  
59  
60  
65  
66  
67  
67  
68

a-  
s.  
t.,  
5,  
i-  
r.  
s.  
t.,  
v.  
c.  
).  
n.  
s.  
k

h  
c.  
s,

r-  
k

n-  
e-  
t,

t.  
h